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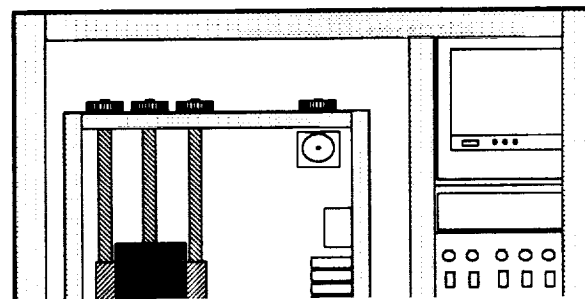
Visualization of Solidification Front Phenomena

by

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and

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16. Abstract		

Directional solidification experiments have been utilized throughout the Materials Processing in Space Program to provide an experimental platform which minimizes variables in solidification experiments. Because of the wide-spread use of this experimental technique in space-based research, it has become apparent that a better understanding of all the phenomena occurring during solidification can be better understood if direct visualization of the solidification interface were possible.

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1.0 Introduction

This research effort studied convection during steady state coupled growth in immiscible systems. Emphasis was placed on hypermonotectic composition alloys. Previous findings have indicated that hypermonotectic alloys can be solidified under conditions which lead to interfacial stability^{1,2,3,4,5}. As a result, it is anticipated that the coupled growth process can be sustained in hypermonotectic alloys in order to produce aligned fibrous microstructures. These structures would contain a higher volume fraction of the hypermonotectic phase than is possible in alloys of monotectic composition. As a result, these hypermonotectic alloys may be quite desirable for several engineering applications.

Unfortunately, convection appears to impede the formation of an aligned fibrous composite structure in immiscible alloy systems^{1,2,3,4}. This problem appears to be primarily due to solutal convection which occurs during the solidification of alloys in the hypermonotectic composition range. This solutal convection appears to either disrupt the solute boundary layer necessary for steady state growth or cause compositional changes in the average composition of the liquid which are sufficient to result in interface instability during directional solidification.

The influence of thermal gradients on the convective stability of a system has been studied since the early 1900's. However, it was not until the early 1980's that the convective stability due to combined thermal and solutal effects during directional solidification was taken into consideration. It is now believed that directional solidification of a hypermonotectic alloy can produce an unstable density gradient in the liquid adjacent to the solidification front which may subsequently cause convection in the system². Furthermore, the resulting convection appears to be capable of disrupting the coupled growth process and subsequently the formation of an in-situ fibrous composite structure.

One way to study this convective instability is through the use of a transparent metal analog system and a specially designed processing facility equipped with an optical imaging system. In this investigation, the transparent immiscible system succinonitrile-glycerol was used and served as a model for studying the general solidification behavior of metallic immiscible systems.

To study convection in immiscible systems, direct observation of the liquid in advance of the solid-liquid interface was carried out during directional solidification. Real-time visualization of the solidification front was accomplished through the use of the modified Convective Flow Analyzer and the transparent metal analog system succinonitrile-glycerol. Using an optical imaging system equipped with transmitted light capabilities, direct observation of the solidification front during processing was relatively straight forward.

The use of transparent organic systems have been proven to show reaction events during solidification which are representative of the events which occur during solidification of metallic samples. These transparent materials have been used to study eutectic solidification⁸, to observe dendritic growth^{9,10,11}, and to model segregation in castings^{12,13}. All of the microstructures and

microstructural transitions observed in monotectic metallic systems have been observed in transparent analog systems containing monotectic reactions ^{6,7,14}.

Cylindrical samples are typically used for directional solidification studies. However, due to the optical requirements of this experimentation and the thermophysical characteristics of the material being utilized (high Prandlt numbers), the sample geometry consisted of a relatively thin plate. A schematic of the sample cell geometry is show in Figure 1. The sample cell was constructed of two 1.6 mm thick glass microscope slides separated by a 1 mm thick Teflon™ spacer. The parallel separation of 1 mm is sufficient to permit natural convective flows in the sample without excessive damping. The sample assembly is held together using silicone sealant around the perimeter edges. Cheri M. Buckhalt at UAB designed and perfected the difficult fabrication techniques for assembling the sample cell. Great care must be taken to avoid exposing the melted succinonitrile-glycerol material to oxygen in the air during the loading process.

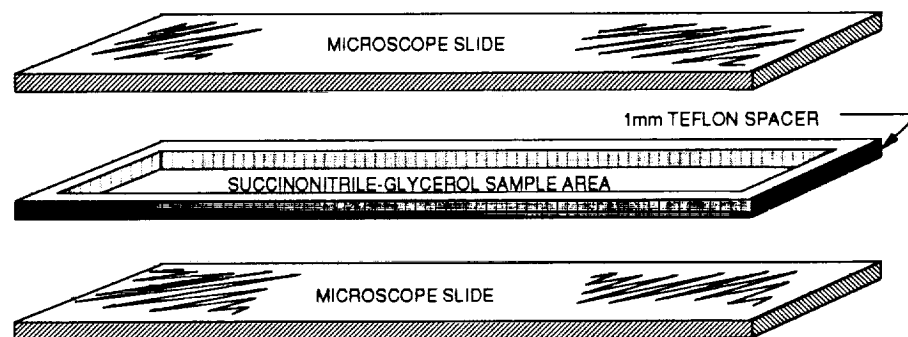


Figure 1: Exploded view of sample cell assembly

One of the major difficulties in carrying out this study was obtaining the proper facilities to process and observe these transparent materials during solidification. Direct visual observation of the solidification front in real-time can greatly reduce the amount of speculation about the interaction between the immiscible phases and convective flows at the interface during directional solidification processing. The Convective Flow Analyzer was originally built under NAS8-36955, D.O. 93. The objective in that study was to monitor the effect of accelerations on fluid flow as the KC-135 flew a parabolic trajectory to simulate various gravity levels. That program was highly successful in that the dampening effect of low g was observed during the transition from 1.8 g to 0.01g using video recording techniques. Consequently, the CFA was very adaptable to being used in this study with some moderate modifications.

2.0 ENHANCEMENT OF THE CFA SYSTEM

There were primarily two main areas of the originally designed CFA which required modification. These included the installation of a translation mechanism to provide directional solidification of the sample and the installation of a high magnification video camera which could image the solidification front. When the CFA was originally built no provisions were made to translate the furnace/cold block assembly in a precisely controlled direction since it was not required for that particular study. In addition the video imaging camera system was designed for a low magnification fixed position application and therefore had to be completely redesigned for this study. Other less involved activities included a new sample holder, increasing the cooling capacity of the cold block and modifying the control and data acquisition software.

With the required low end translation parameter of 0.1 microns/sec the translation mechanism had to be fabricated using the highest quality materials available and the strongest structural designs possible. Three precision ball screw drives with class C0 tolerances were purchased from THK Company. According to the manufacture this highest class of tolerance provides an accumulated reference lead error of ± 4 microns over a distance of 300 millimeters. During actual experiment runs the total translational distances were generally limited to 10 millimeters of distance. The accuracy of the lead over that short of a distance was therefore ± 0.133 microns or ± 0.013 microns/mm. The three ball screw drives were arranged in a triangular relationship between the upper and lower plate experiment rack framework. Class C2 thrust bearings were used to support the ball screw drives. The allowable compression/tension loading for each 16 mm diameter drive is conservatively rated at 2100 kgf. A 0.75 inch thick triangular shaped aluminum platform onto which the sample cold block, furnace and camera assembly was mounted and supported at each corner by the three drives. Power transmission to the three drives was accomplished using a system of sprockets, a timing belt and belt tensioners to remove slack at all points from the belt. The lead of the three ball screw drives is 5 mm per revolution.

To drive the translation system a Compumotor CX microstepping motor coupled to a 60 to 1 gear reduction was used. This particular system, with the motor capable of 12,800 microsteps per revolution, provided a range of translation rates from 0.08 microns/sec to 2 cm/sec. Since the motor is digitally controlled errors associated with other analog based systems (servos) was eliminated. This provided an extremely accurate and reproducible method of translation and positioning. The positioning accuracy of the stepper motor itself is ± 5 arc minutes or ± 3 microsteps which translates to ± 0.019 microns in translational distance. The repeatability of the motor is ± 5 arc seconds or ± 0.01 microsteps which translates to ± 0.000065 microns in translational distance. Due to inherent high accuracy of this motor, the position data displayed on the CRT during experiment runs is calculated from the number of pulses sent to the stepper motor. It was determined that this method of monitoring would be far more precise and accurate than any presently available analog based position sensor over the range of translation distances expected. Tests were performed on the

position accuracy when the modifications were completed. Using a precision digital micrometer, as a standard reference, agreement between the computer display and the micrometer was within the measuring accuracy of the micrometer itself which was 1 micron.

The next step in the modification process was to develop a high magnification video imaging system which could perform the following:

- ① met the requirement of being able to image particles as small as 1 micron in diameter,
- ② viewing could be positioned in all three axes, and
- ③ was rock solid and did not vibrate or flex under the extremes of the KC-135. This proved to be the most difficult part of the modification process.

The Titan Tool Supply Company lens assembly was built up using a 1X sub lens, 2.0X video adapter, 90° optical path bender, and the micro video variable zoom objective lens. This provided a magnification range of 19X to 121X when displayed on a 9 inch diagonal video monitor and a resolution of 57 to 128 line pairs/millimeter. A 2X sub lens was experimented with in the prototype system however due to physical size constraints, could not be used as the camera/lens assembly had to be positioned 10 inches from the sample. This would have made the task of designing and building the camera mount beyond reason. Using this lens could have provided a maximum magnification of 242X. As result of using the 1X sub lens the smallest particle that could be imaged was limited to 5 microns.

To provide the three axis positioning capability for the camera three Newport model 423 low profile positioning stages were adapted. A special set of robust mounting plates and angle brackets were machined to provide the overall mounting system. After the first KC-135, flight improvements had to be implemented to tighten up the system as some minor flexing could be detected as the aircraft flew in and out of low gravity. Figure 2 provides details as to the camera mount assembly.

During the initial design process for the modifications to the CFA there existed some uncertainty as to the proper design of the hot and cold block. As result, time was devoted to thermodynamically modeling in three dimension this part of the CFA in order to best determine the shape and dimensions. To characterize the CFA cold/hot block system, a finite element model was analyzed for various configurations using the software program Algor. The model consisted of an aluminum hot block, borosilicate glass sample holder and copper cold block. The temperature conditions were added to the perimeter of the hot and cold block and comparison were made between the various configurations. There were many factors to examine in order to attain the proper temperature gradients. These factors included the contact area of the hot and cold block, the gap between the hot and cold block and the temperature settings of both blocks. The model was drawn and temperatures added using Algor's Superdraw II version 3.16. The model was then decoded using the Thermal Decoder version 1.05 and verification of the mesh was done in the Thermal Superview version 1.08. Once a proper mesh was achieved, the model was analyzed in the Steady-State Heat Transfer Analysis version 10.00. The final product was dithered in the Thermal Superview for characterization of the system. Figures 3 and 4 provide the visual indication of

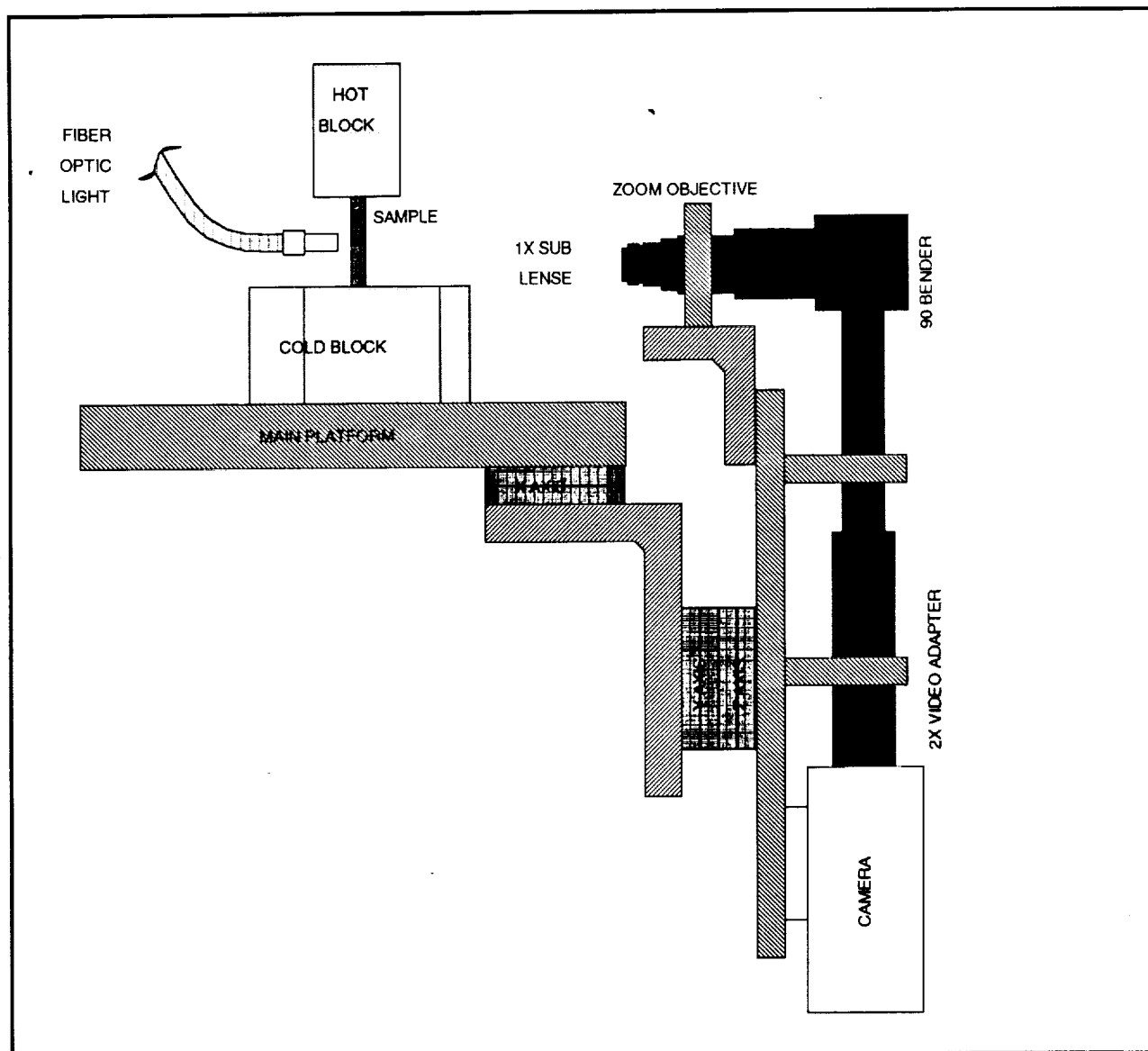


Figure 2: Side view of camera mount assembly.

the temperature gradient across the sample. It was determined from the analysis that a 0.375 inch cold block contact area on the sample would be sufficient to generate the required thermal gradient. While the model indicated a non planar isothermal condition between the cold and hot blocks, in reality the actual solidification front in the all samples was almost perfectly flat. The reason for the discrepancy is believed to be due to the fact that the sample material of succinonitrile-glycerol and the Teflon seal was not included in the model. These materials would provide a higher thermal conductivity through the system than glass alone.

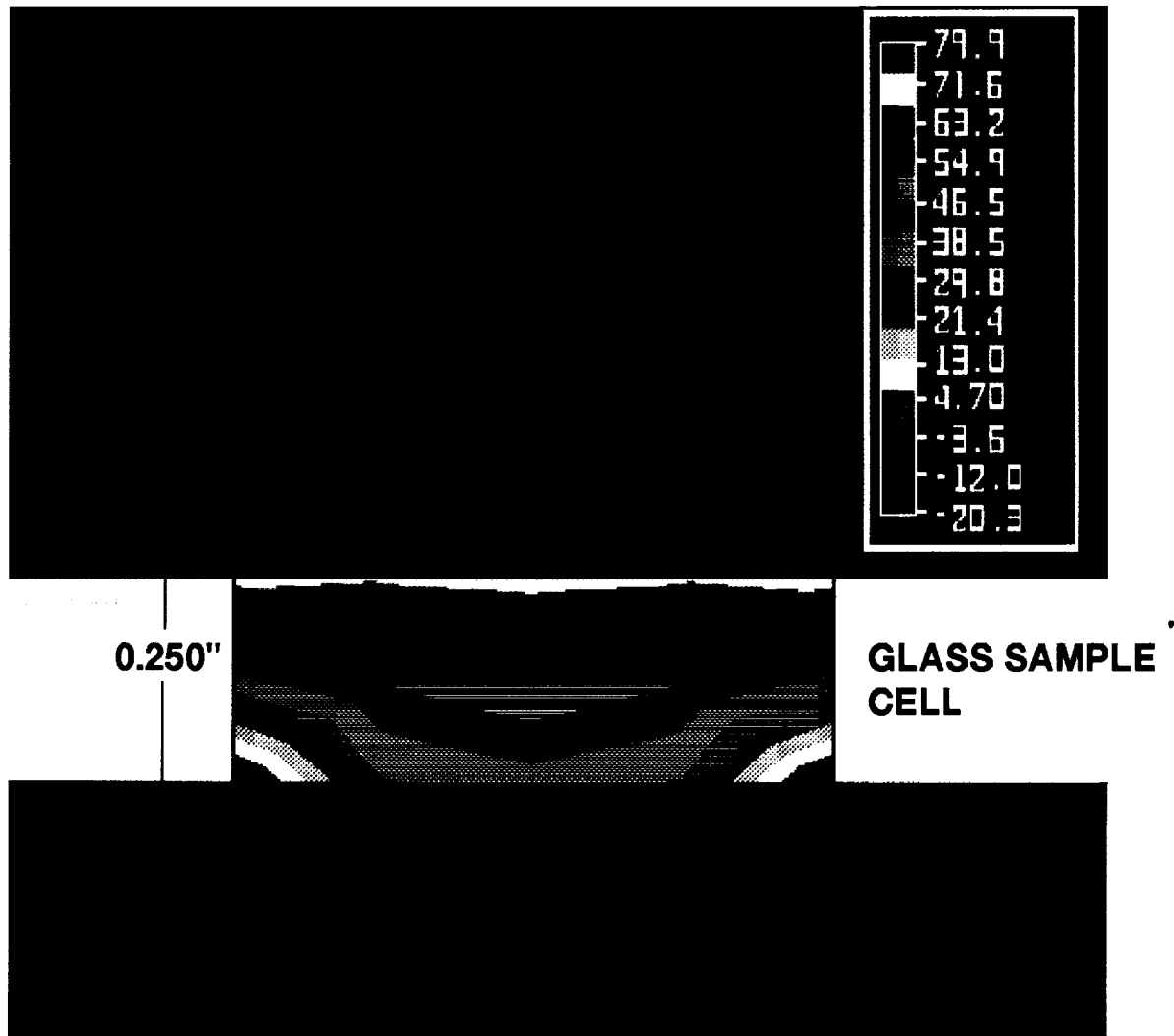
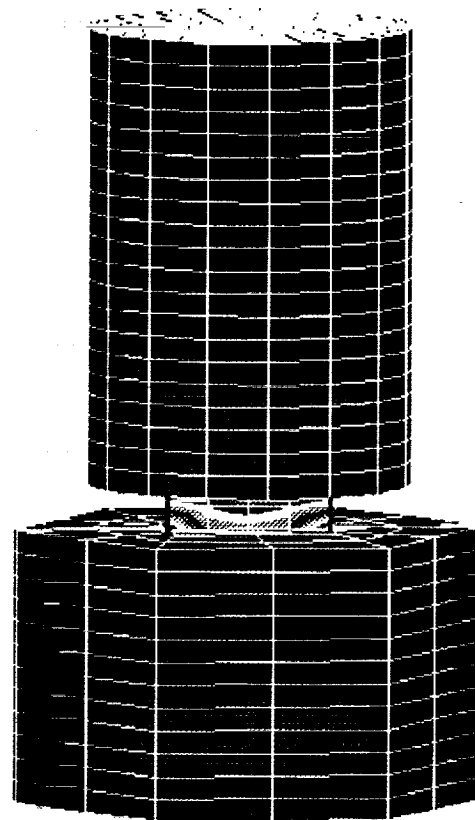


Figure 3: Finite element model of hot block-sample-cold block thermal characteristics. Contact area of the cold block with the sample was determined to be 0.375" optimum



Temperature plot of isotherms

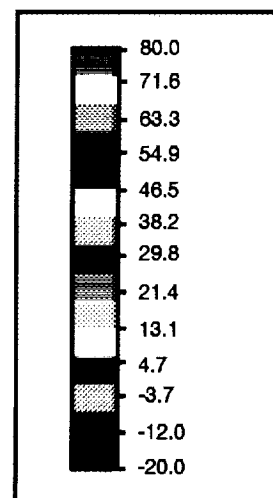


Figure 4: Three dimensional view of Figure 3.

Based on the finite element model the hot and cold block were fabricated to the required specifications and then replaced the existing hot and cold blocks in the CFA. Attachment of the six thermoelectric cooling elements was performed using silicone adhesive. This provided a reliable mounting and good thermal contact with the cold block. In order to achieve a -40°C temperature of the cold block a supplementary cooling unit FTS Systems, Inc model RC-25-LT was installed in line. This recirculating cooler used R-12 freon in a tube-in-tube heat exchanger to cool down the ethanol/10% Prestone™ coolant. The temperature controller for the chiller was usually set to 10 degree differential above the setpoint for the cold block. In this configuration the stress on the six thermoelectric modules was kept to a minimum. Total heat pumping capacity for the six modules was over 300 watts. Temperature control of the cold block was maintained within 0.10°C by using a Eurotherm model 808 digital controller with a platinum RTD temperature sensor. The furnace and associated hot block was controlled using an identical temperature controller and RTD to provide the same 0.10°C resolution and accuracy.

3.0 RESULTS

Prior to the first flight of the modified CFA, on the KC-135 in December of 1992, sufficient ground based runs were performed to confirm the operational capability and characteristics of the system. Minor changes had to be performed to the software to better accommodate the required control parameters of the translation system; however, no others actions were required prior to the first flight. The software was developed to make the operation of the system as simple as possible. This proved highly valuable during actual KC-135 flights. Proper operational techniques were determined and rehearsed to better guarantee good results during future flights. The only difficult part in the operation of the CFA is in the loading of the glass sample cell. It was soon learned that these cells could be easily broken during the loading process, however, sufficient practice reduced the incidence of failure to nearly zero.

Finally a test article safety data document was prepared and submitted to MSFC and JSC Safety on the added FTS Systems recirculating cooling system. This was to be the first time this particular item had flown on the KC-135 and as such had to be qualified for flight. No problems were experienced during the qualification process.

One of the major objectives of this study was to investigate the theory that convective instability occurs within the boundary layer which is formed during directional solidification. Tiller experimentally confirmed the existence of this boundary layer, and it has been speculated that convective flow within this boundary layer has a strong dependence on gravity level. Many authors have performed theoretical linear stability analyses in an attempt to understand this convective instability; however, convective instability within the boundary layer has not yet been experimentally evaluated much less observed in-situ during directional solidification. More precisely stated, the crux

of this study was intended to confirm the dependence of gravity level on the convective flow within the boundary layer through direct observation during directional solidification.

Directional solidification experiments were performed aboard NASA's KC-135 aircraft to produce the needed change in gravity levels. Three separate weeks of flights were scheduled for this study, and a total of seven samples were processed successfully. In all of these flights, the directional solidification experiments were performed using a thermal gradient of approximately 70°C per 0.25 inch and a translation rate of 0.1 microns/second. The high thermal gradient was utilized in conjunction with a relatively low growth rate to yield a thermal gradient to growth rate ratio (G_L/V) which satisfied the constitutional supercooling criterion. With this condition satisfied, constitutional supercooling was avoided, and thus the formation of hypermonotectic L_2 droplets in advance of the growth front was suppressed and the growth front remained planar.

Directional solidification experiments were also performed under normal 1-g conditions as the flight samples using the same G_L/V ratio to avoid constitutional supercooling and produce a planar growth front.

For both flight and ground based samples four compositions were utilized. These compositions included 2 wt% hypomonotectic, 1 wt% hypermonotectic, monotectic and 1 wt% hypomonotectic. Cheri Moss, graduate student at UAB, was responsible for all sample production and analysis of the results in fulfillment of her masters thesis requirements.

Each sample was labeled with an identification code. For example, sample 8.5A1211 had a composition of 8.5 wt% glycerol (SCN-8.5wt%G) and the alloy was produced on December 11. For each composition, a flight sample (alternating high and low gravity conditions) was processed and a ground sample (constant 1-g level) was processed for comparison purposes.

For each composition, the conditions for interfacial stability were achieved, and the effect of gravity level on the convective stability of the system during directional solidification was evaluated. Events at the growth front during processing were recorded with the S-VHS format VCR to aid in analysis of convective flow which occurred.

3.1 Flight Results

First KC-135 flight

The first flight tests for the modified CFA system occurred during the week of December 14-18, 1992. Four samples were to be processed with two more as backups, however, only two samples were processed during this week of flights. The KC-135 was grounded two out of the four days due to mechanical difficulties.

The two samples 8.4AP1211 and 9.5A1211 were directionally solidified under the alternating gravity level conditions aboard the aircraft. Sample 8.5AP1211 was the very first sample processed in the CFA aboard the KC-135. In this sample code, the letter P meant that the alloy contained approximately 10 wt% polystyrene spheres. These spheres were intended as a tracer to aid in

visualizing convective flows in the boundary layer during processing. These spheres had a diameter of 10 microns $\pm 3\%$. Although the spheres were found to be neutrally buoyant in the alloy, they did not follow or trace the fluid motion. Some of the spheres adhered to the cell walls, some were clumped together and a few were actually following the fluid motion. As a result, it was somewhat difficult to depend on the spheres to trace the motion of the fluid accurately.

A promising observation was recorded during directional solidification of 9.5A1211. While processing the sample, what appeared to be a boundary layer began to form at the solid-liquid interface during the low-gravity portions of flight. Then, during the high-gravity portion of flight, convective flow swept away this boundary layer. This boundary layer had a different contrast from the bulk of the fluid due to composition differences between the boundary layer fluid and the bulk fluid. This composition difference is predicted from the directional solidification theory. Figures 5, 6 and 7 were captured off the video tape using a video frame grabber and then printed out on a high resolution laser printer. Figure 5 is of the sample during the low-g period and shows the build up of the boundary layer. Figure 6 is the same sample after just entering high-g and the disruption of the boundary layer occurs rapidly. This was an expected result, however, there appeared to be a noticeable amount of fluid motion back-and-forth across the sample cell. This fluid motion made it somewhat difficult to determine if the flow which disrupted the boundary layer was driven by convective instability alone or in some part due to the back-and-forth motion of the fluid. It was speculated that this anomalous motion was due to the fore-and-aft acceleration of the aircraft during parabolic flight. Figure 7 is of the same sample composition (9.5wt% glycerol or 2% hypermonotectic) and was processed as a ground based sample. As was expected, convective cell flow was predominant all along the solid/liquid interface.



BOUNDARY LAYER



Figure 5: 2% hypermonotectic sample in low-g. Note the formation of a boundary layer.

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Figure 6: Same 2% hypermonotectic sample just after entering high-g.

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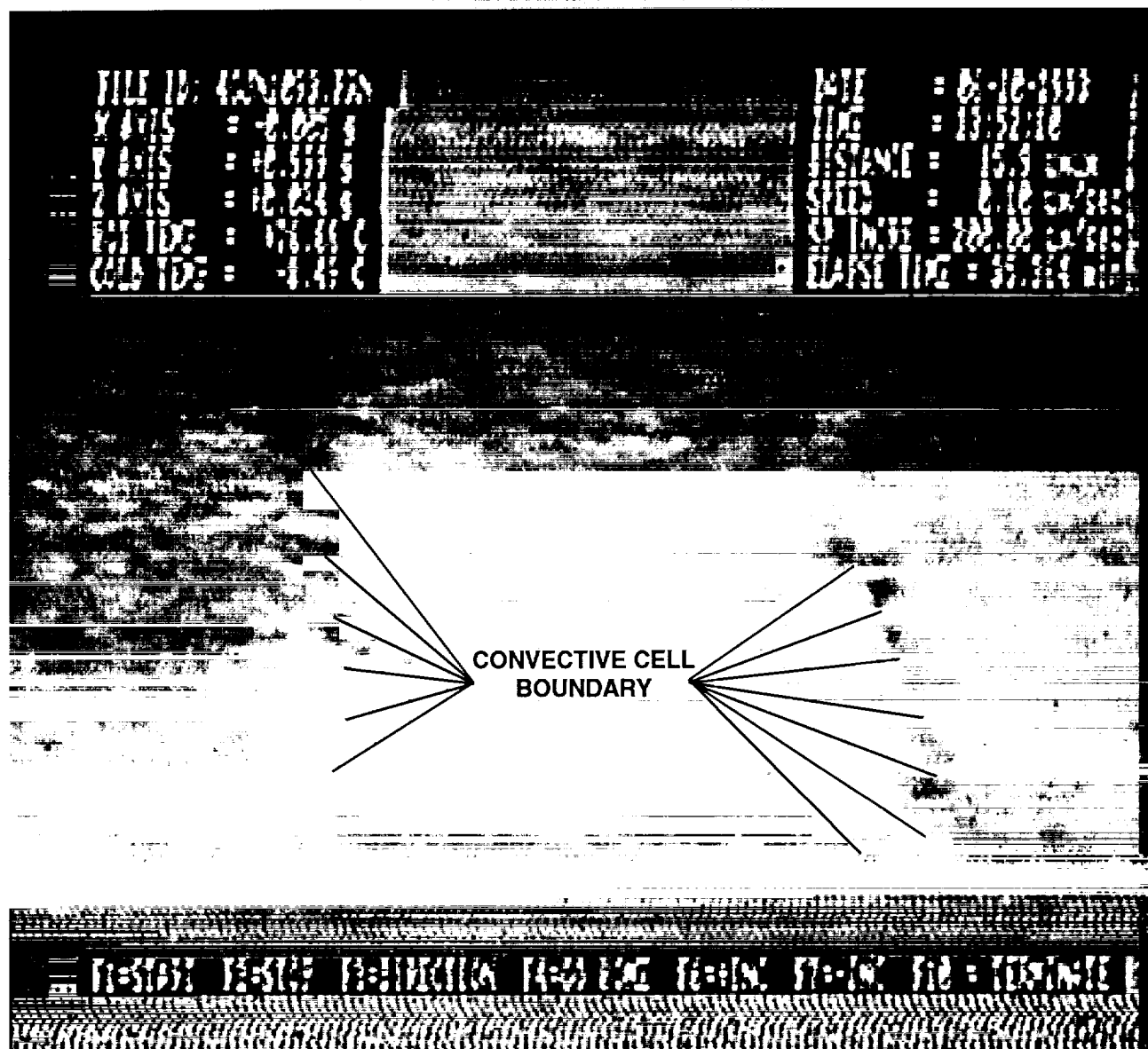


Figure 7: 2% hypermonotectic sample in one-g.

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Second KC-135 flight

During the week of January 11-15, 1993 four samples were to be processed, however, only two were completely successful. Attempts to directionally solidify 9.5A0108 and 7.5A0108 were futile. During processing of 9.5A0108 it was determined that the sample came loose from the sample holder and as result the data was not usable. Equipment failure prevented the directional solidification of 7.5A0108. The translation system of the CFA was disabled due to a blown fuse in the power supply. This did not allow translation of the sample through the furnace. However, the sample was loaded into the furnace anyway and a temperature gradient was imposed on the sample. Observations of the liquid in advance of the solid-liquid interface during flight confirmed that there was a noticeable amount of motion to the left and right across the sample which was independent of the directional solidification process. Earlier speculation of the fore-and-aft acceleration of the aircraft causing the anomalous fluid motion was reinforced from observations of this sample during flight. Examination of the gravity level data while the sample was in a static mode determined that there was a traceable amount of fore-and-aft acceleration of the aircraft. In an effort to minimize the influence of this fore-and-aft acceleration on the fluid inside the sample, the sample orientation relative to the fore-and-aft axis was modified so as to place the long face of the sample perpendicular to the fore-aft axis of the aircraft.

Two samples, 8.5A0108 and 8.5BP0108, were directionally solidified under the alternating gravity level conditions aboard the aircraft. Both sample showed the build up of the boundary layer during low gravity portions of the flight and the convective flow disrupting the boundary layer during the high-gravity portions of flight. Just as with 9.5A1211, the boundary layer seen in 8.5A0108 and 8.5BP0108 was distinguished from the bulk of the liquid due to composition differences. Again these were expected results for convective stability in 0.01 g levels and convective instability for 1.8 g levels. However, for both samples, there was still a noticeable amount of fluid motion in advance of the growth front due to the fore-and-aft acceleration of the aircraft. It was somewhat difficult to differentiate the flow which disrupted the boundary layer due to convective instability and the flow attributed to the acceleration of the aircraft. Therefore, it could not be concluded that the disruption of the boundary layer was a result of fluid flow driven by convective instability alone.

Something which could be determined from these two samples was the usefulness of the polystyrene spheres to trace fluid flow. Since 8.5A0108 and 8.5BP0108 were the same composition, it was easy to evaluate the benefit of using the spheres as a tracer material. As previously discussed, the spheres did not effectively follow or trace the fluid motion. It was decided from processing these two samples that there was enough contrast in the fluid in advance of the growth front to visualize convective flows without the spheres. As a result, samples for subsequent flights did not contain these spheres.

Third KC-135 flight.

During the week of March 22-26, 1993 four more samples were processed successfully. For all samples processed during this week, the samples were reoriented as discussed previously to minimize the effect of the fore-and-aft acceleration of the aircraft on the fluid during processing. To accomplish this task, the entire CFA using was mounted on a secondary base plate at an angle such that the minimum sample dimension was perpendicular to the fore-and-aft axis of the aircraft.

Efforts to directionally solidify 8.5A0319 (fifth sample) failed. While lowering the hot zone around the sample to establish the thermal gradient, one of the glass slides cracked and some of the alloy leaked out of the sample. Subsequent processing could not be completed.

Sample 9.5A0319 was successfully processed under the alternating gravity level conditions aboard the aircraft.. As with previous samples successfully processed, there was a build up of the boundary layer during low gravity. Then during the high gravity periods the convective flow disrupted and swept away the boundary layer.

With the improved orientation of the sample relative to the fore-and-aft axis of the aircraft, the fluid motion attributed to the acceleration of the aircraft was virtually damped out. With this in mind, it can be said with some certainty that the fluid flow seen during the high gravity periods was due to convective instability.

As with 9.5A0319, the fluid motion attributed to the acceleration of the aircraft was virtually damped out with the improved orientation of the sample, and again, it can be said with some certainty that the fluid flow seen during the high-gravity portions of the flight was due to convective instability of the system. Sample 7.5A0319 was subsequently directionally solidified. Unlike the previous samples which were successfully processed, there was not a distinct build up of the boundary layer during the low-gravity portions of flight. Consequently, there was no driving force for convection in the fluid in advance of the solidification front during the high-gravity portions of flight. This result is in agreement with directional solidification theory concerning an alloy of monotectic composition. Upon directional solidification of an alloy of monotectic composition, the reaction which occurs is the decomposition of one liquid, L_1 , into a solid, α , and another liquid, L_2 . Under conditions of interfacial stability, the monotectic reaction transforms L_1 into a fibrous or rod-like structure of alternating α and L_2 . The formation of this fibrous microstructure during directional solidification of a monotectic composition alloy has been experimentally confirmed for numerous immiscible alloy systems. Unfortunately, after returning to Huntsville for some unknown reason the video tape of this run was blank and no visual record therefore exists.

Sample 6.5A0319 was also processed successfully during flight. Similar to 7.5A0319, there was no formation of the boundary layer during low-gravity conditions and subsequently no convective flow in the liquid during high-gravity.

During the month of July of this year all required ground based samples in support of the flight runs were completed and Cheri M. Buckhalt is still in the processing of analyzing those results.

In addition, a thermal gradient run was successfully completed. In this case a glass sample cell was instrumented with two 0.010" diameter type K thermocouples and the sample cavity filled with succinonitrile-glycerol. The data acquisition software was modified to allow the collection of two more temperature channels. The sample was loaded into the CFA system and processed at a directional solidification rate of 0.1 microns/second and the same temperature settings used on all previous samples. Data was collected by the computer over a period of three hours. Those results are to be published in Ms. Buckhalt's thesis.

4.0 ACKNOWLEDGMENT

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